Process Specification for Pickling, Etching, and Descaling of Metals

Engineering Directorate

Structural Engineering Division

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REVISIONS		
VERSION	CHANGES	DATE
--	Original version	2/03/00
A	Changed Division name. Corrected errors in metal removal thickness before penetrant inspection in Sections 3.0 and 6.3.2. Re-titled and revised Section 6.1.	10/2004
B	Extensive Changes in sections 6, 7, and 8, associated with the processing of titanium.	12/2004
C	Retitled Section 4.0 (Applicable Documents) to (References) and updated document titles and numbers. Reformatted tables in Section 6.3 (Special Requirements),	2/2007

1.0 SCOPE

This document provides the standard requirements for etching, pickling, and descaling of metals by acids or alkali aqueous solutions.

It does not cover the light pickling of Monel to remove embedded foreign metals, which is covered by the passivation PRC.

2.0 APPLICABILITY

This specification shall be applicable whenever a pickling, etching, or descaling is invoked per Section 3.0, "Usage". It is also in force whenever shop procedures that meet this specification are invoked by another process specification or shop instruction.

3.0 USAGE

This specification covers a wide variety of processes and metals, so the engineering drawing callout shall be specific enough to ensure that the proper work instructions are used.

The term "Pickling" is usually used to describe a process that removes surface oxides (scale) and removes contamination from surfaces that are oxidized or corroded. The term "Etching" is usually used to describe a process that removes parent metal in a controlled manner, sometimes to achieve specific surface textures. "Descaling" is usually used to remove scale or heat tint caused by heat treating or welding. However, the terms are often used interchangeably.

The following drawing callouts are for standard JSC processes.

Stainless Steels
(including CRES 300 series except for 303, 15-5PH, A286, and Custom 455)
DESCALE TO REMOVE EDM RECAST LAYERS PER NASA/JSC PRC-5010.
ETCH FOR PENETRANT INSPECTION PER NASA/JSC PRC-5010.
DESCALE TO REMOVE HEAT TINT PER NASA/JSC PRC-5010.

Nickel Alloys
(none)

Titanium (6Al4V)
DESCALE TO REMOVE EDM RECAST LAYERS PER NASA/JSC PRC-5010.
DESCALE TO REMOVE HEAT TINT PER NASA/JSC PRC-5010.

Aluminum (Most common alloys)
ETCH FOR PENETRANT INSPECTION PER NASA/JSC PRC-5010.

All new pickling, etching and descaling processes shall be qualified for the alloy being processed. Once a process is qualified, the qualification testing does not have to be repeated for that alloy if no essential process variables have changed and no processing problems are discovered. The details of the qualification will be recorded in a Process Qualification Record. Limitations of the process shall be noted in the PQR. Process Qualification Records will be made available on the ES home page for all qualified processes.

Pickling, etching and descaling processes for other metals and other purposes can be qualified when necessary, but design should consult with an ES4 materials engineer to ensure that a procedure can be developed and successfully qualified before manufacturing needs the process.

A significant amount of material may be removed during pickling, etching, or descaling processing. In most cases, this will be compensated for during machining, but there may be some cases for which dimensional control may be difficult to maintain. The typical maximum metal removal after etching aluminum and titanium for penetrant inspection is .0006 inches and for stainless and nickel-based alloys are .0004 inches.

4.0 REFERENCES

The following documents were used in developing this specification:

SOP-007.1	*Preparation and Revision of Process Specifications*
JPR 8500.4	*Engineering Drawing System Manual*

The following documents are called out as an extension of the requirements given in this specification. All documents listed are assumed to be the current revision unless a specific revision is listed.

ASTM A380	*Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems*
ASTM B600	*Descaling and Cleaning Titanium and Titanium Alloy Surfaces*.
NASA/JSC TI-5000-01	*Metal Finishing Technicians, Training Instruction for*

5.0 MATERIAL REQUIREMENTS

N/A

6.0 PROCESS REQUIREMENTS

6.1 WORK INSTRUCTIONS

All work shall be performed to written procedures. The work instructions shall contain sufficient detail to ensure that the manufacturing process produces consistent, repeatable products that comply with this specification.

For work performed at JSC facilities, these work procedures consist of Detailed Process Instructions (DPI's).

For contracted work, the contractor shall be responsible for preparing and maintaining, and certifying written work procedures that meet the requirements of this specification.

6.2 GENERAL REQUIREMENTS

Pickling, etching, and descaling processes shall be controlled to effectively remove oxidation or damage on metal surfaces while minimizing any deleterious effect on the surface. The surface finish shall be smooth, without pitting or attack of grain boundaries. For susceptible metals, the process should not have a negative effect on the mechanical properties of the metal.

General requirements for bath compositions are listed in the sections for each special process. The specific compositions and operating parameters of each process shall be detailed in the work instruction and summarized in the PQR. Work instructions for stainless steels and alloys shall be in compliance with the standard practices of ASTM A 380. Work instructions for titanium alloys shall be in compliance with the standard practices of ASTM B 600.

Free machining stainless steels shall not be pickled, etched, or descaled.

While descaling or removing the EDM layer on titanium parts, the surface may become passive and resistant to further chemical cleaning. If this occurs, the part shall be blasted with abrasives to reactivate the surface and the part returned to the processing solution. Allowable abrasives and maximum sizes are #13 glass bead, 220 grit alumina, and 220 grit silicon carbide.

High temperature caustic solutions may be used to precondition titanium prior to acid treatments. This treatment may remove all visible scale or oxidation, but a flash pickle afterwards is required at a minimum.

Oxygen and nitrogen diffuse into the titanium at high temperatures and form a brittle phase called alpha case, which can readily fracture and subsequently initiate several types of service or test failures. The visual inspection of titanium parts after pickling or descaling shall include verification that alpha case has been removed.

6.3 SPECIAL REQUIREMENTS

6.3.1 ETCHING FOR SUBSEQUENT METAL FINISHING

Etching processes used as preparation of metals surfaces for subsequent metal finishing shall use process solutions controlled and qualified by the subsequent metal finishing process and its work instructions.

6.3.2 ETCHING FOR PENETRANT INSPECTION

When metal parts are etched for penetrant inspection, a minimum layer of metal must be removed to meet NDE reliability requirements. For aluminum and titanium, the minimum metal to be removed is .0004 inches. For stainless steel and nickel-based alloys, the minimum metal to be removed is .0002 inches.

Process control coupons shall be used to verify the minimum metal removed.

Alloy	Process Solution	Temperature	Notes
Stainless Steels	Nitric Acid HF Acid	Ambient	Concentrations per ASTM A380
Nickel Based-Alloys (other than Monel)	TBD		
MP35N	TBD		
Monel 400 and K500	TBD		
Titanium	NaOH 40-50%	240-280F	Descaling pretreatment
Titanium	Nitric Acid 7% HF Acid 3%	Ambient	Total process time shall be no longer than 30 minutes.
Titanium	Nitric Acid 20-29% HF Acid 1.5-2.5%	Ambient	10:1 ratio or greater
Aluminum	NaOH 5%	Ambient	

6.3.3 DESCALING TO REMOVE EDM RECAST LAYER

Qualification coupons shall be processed that verify that the recast layer will be completely removed. The EDM cutting schedule used in qualification shall have a cutting current (usually denoted as IP) that is the same or higher than the production parts.

Alloy	Process Solution	Temperature	Notes
Stainless Steels	Nitric Acid HF Acid	Ambient	Concentrations per ASTM A380
Nickel Based-Alloys (other than Monel)	TBD		
MP35N	TBD		
Monel	FeCl3 HCl		
Titanium	NaOH 40-50%	240-280F	Descaling pretreatment
Titanium	Nitric Acid 7% HF Acid 3%	Ambient	Total process time shall be no longer than 30 minutes.
Titanium	Nitric Acid 20-29% HF Acid 1.5-2.5%	Ambient	10:1 ratio or greater
Aluminum	Phosphoric Acid 80% Acetic Acid 15% Nitric Acid 5%	212°F	

6.3.4 DESCALING TO REMOVE SCALE AND HEAT TINT

When parts are to be descaled to remove scale or heat tint, the metal parts shall be immersed in the process bath for as long as necessary to remove the oxides, up to the maximum time allowed by the work instructions. Usually the dimensional changes are uncontrolled. Qualification coupons shall be processed at the maximum time allowed by the work instructions.

The compositions of the descaling process solutions shall be as follows:

Alloy	Process Solution	Temperature	Notes
Stainless Steels	Nitric Acid HF Acid	Ambient	Concentrations per ASTM A380
Nickel Based-Alloys (other than Monel)	TBD		
Monel	$FeCl_3$ HCl	Ambient	
Titanium	NaOH 40-50%	240-280F	Descaling pretreatment
Titanium	Nitric Acid 7% HF Acid 3%	Ambient	Total process time shall be no longer than 30 minutes.
Titanium	Nitric Acid 20-29% HF Acid 1.5-2.5%	Ambient	10:1 ratio or greater
Aluminum	NaOH 5%	Ambient	

7.0 PROCESS QUALIFICATION

The work instructions for each process shall be qualified on non-flight hardware or coupons before the flight hardware is subjected to the process. The processes shall be individually qualified for each alloy and are valid only for a specific alloy or processing facility. Each process shall be evaluated with qualification coupons to determine adherence to the general requirements for pickling, etching, or descaling and for any special requirements. Once a process is qualified, the testing does not have to be repeated if no essential process variables have changed and no processing problems are discovered.

Process qualification results shall be recorded on a Process Qualification Record (PQR) and signed by the metal finishing supervisor and an engineer from ES4, Materials and Processes Branch. The PQR shall include, as a minimum, the alloy processed, the process solution compositions, the essential processing variables, and the results of the examinations.

Approved PQR’s shall be stored on the ES Home page.

8.0 PROCESS VERIFICATION

Each part shall be inspected after processing to verify that the surface finish is smooth, free from oxide, scale, pitting or grain boundary etching.

Process Control coupons shall be used to determine the etch rate when the surface will be etched before NDE inspection. These coupons shall be made of the same alloy as the part being processed. The verification coupons shall be thin and flat to maximize the differential thickness measurement. Verification coupons shall be etched and the thickness changed determined on the day of processing before the production work is processed. The bath’s etch rate shall be determined and the etch time adjusted to guarantee the minimum metal removal is achieved on that day.

Records of self-inspection and MIPS shall be kept as quality assurance records.

8.1 ADDITIONAL REQUIREMENTS FOR TITANIUM

Titanium parts shall be visually inspected to verify that alpha case has been removed.

Process verification coupons to verify freedom from hydrogen embrittlement are required when titanium parts are processed with nitric acid/hydrofluoric acid solutions.

Tensile coupons are required when the Nitric Acid 7% -- HF Acid 3% solution is used. The coupons shall be made of the same lot of material as the production parts. Coupons for EDM layer descaling process hydrogen verification shall have their gage section cut by the EDM process. Coupons for NDE etch process hydrogen verification shall be conventionally machined (unless the part is EDM machined). Tensile coupons made by the EDM process shall be rectangular sheet type (Figure 1 of ASTM E8) and shall have the same thickness as the minimum section of the part or 0.300 inches, whichever is less. The preferred coupon for conventionally machined parts is the ASTM E8 cylindrical-type and shall have a diameter less than the minimum section of the part or 0.500 inches diameter, whichever is less. Tensile coupons shall be tested to ASTM E8, except that coupon shall be chilled to 32°F immediately before testing. Hydrogen verification tensile test coupons made of 6-4 titanium shall have an elongation greater or equal to 10.0% and a reduction of area greater or equal to 25%.

Tensile coupons are not required when a solution with 10:1 or greater ratio of nitric acid to hydrofluoric acid is used. Instead a total hydrogen pickup test may be performed on a daily or lot basis. The coupon for this test shall be processed, at the minimum, the maximum time the coupon is immersed in the acid solution. The coupon is not required to be from the same lot of titanium, but it does have to be the same alloy and heat treatment. The details of this test and the calibration of the equipment shall be documented in the facility work instructions for the process.

9.0 TRAINING AND CERTIFICATION OF PERSONNEL

Training requirements for metal finishing technicians shall be written to cover the requirements of this specification. Trainee shall be certified following the successful working under the supervision of the metal finishing facility manager. Training and certification records shall be kept.

For work performed at JSC facilities, these requirements shall be satisfied by the training and certification of personnel per TI-5000-01 Training Instruction.

10.0 DEFINITIONS

Mandatory Inspection Point (MIP)	A second-party inspection process designated during a manufacturing operation.
PQR	Process Qualification Record.